PROJECT NUMBER: 2520
PROJECT TITLE : Organic Synthesis
PROJECT LEADER : Y. Houminer
PERIOD COVERED : July, 1991

I. FLAVOR RELEASE TECHNOLOGY

A. **Objective:** To investigate the synthesis and pyrolysis of various flavor release systems for use in new or improved products.

B. **Results:** A series of experiments were conducted with PolyMIC to determine effective levels for platicizers. PolyMIC was slurried in abs. ethanol to which was added the target level of the test platicizer. Material was produced with triacetin and triethyl citrate at 5 and 10%. After removal of the solvent, the product was a free flowing powder in all cases. Each of the treated PolyMIC samples showed a lower melting point, approximately 10 and 20°C for 5 and 10% respectively. The results of the these experiments were given to W. Nichols who conducted a 2 pound experiment with triacetin at 10%. The resulting material was successfully applied as a hot melt to cigarette paper during cigarette manufacture.

The reaction of α-hexylcinnamaldehyde and cinnamyl isobutyrate gave the desired β-hydroxyester. A small sample was purified and was shown to undergo smooth pyrolysis at 300°C to give the corresponding aldehyde and ester.

Studies on the acetalation of α,α-trehalose with α-hexylcinnamaldehyde continued this month. Preparative HPLC isolation of products from the reaction of trehalose and α-hexylcinnamaldehyde dimethyl acetal yielded fractions which contained significant quantities of α-hexylcinnamaldehyde and some trehalose. It was concluded that breakdown of the desired products was taking place on the silica HPLC column.

CR-2950 was incorporated into two different cigarette papers at a level of 1.5 - 2.0% by Ms. S. Tafur. Handmade cigarettes were placed in desert and jungle rooms for 4 days. Analysis of the cigarette papers by a reversed phase HPLC procedure showed no difference between the amount of CR-2950 on paper of the test cigarettes and the control.

Hydrolysis of menthyl 3,4:5,6-di-O-isopropylidene-D-gluconate was carried out under two different conditions. We have isolated menthyl 3,4-O-isopropylidene-D-gluconate, which on pyrolysis via a 220° GC injector port gave significant amount of menthol. We are continuing our effort to isolate menthyl D-gluconate.

In an attempt to prepare bis-(3-oxo-α-ionyl)carbonate through the intermediate 3-oxo-α-ionyl chloroformate, three compounds were isolated and identified, none being the desired product. Two of the compounds were identified as Z- and E-megastigma-4,6,8-trien-3-one, and the other as 3-oxo-α-ionyl chloride. All three compounds are decomposition products of 3-oxo-α-ionyl chloroformate.

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II. FILTER MATERIALS AND PAPER TECHNOLOGY SUPPORT

A. **Objective:** To find outside suppliers for large quantities of new filter materials and inorganic paper additives.

B. **Results:** Jerry Johnson and C. Woodings of Courtaulds visited the center and extensive discussions were held covering current work and samples status. New handsheets have been made from Courtaulds partially deacetylated cellulose and yielded filters with acceptable RTD. These will be attached to cigarettes and subjective evaluation conducted shortly. Discussions with Courtaulds have indicated that the problems with dispersion of the cut CA is probably due to the finishing agents. Plans have been formulated to evaluate alterant agents to allow for effective dispersion.

Trials conducted at Nina mill have shown that Courtaulds' fibrids can be effectively incorporated into CA and CA/soft wood mixtures to give acceptable paper. Problems were encountered with craping of the paper. Discussions were conducted with Courtaulds on this and an additional 50 kg of fibrids has been requested for trials.

III. MISCELLANEOUS

Synthesis of delta-Cadinene: 4-Methylanisole was reduced by lithium/ammonia to give 4-methyl-3-cyclohexenone. This was converted to the pyrrolidine enamine for reaction with 6-methyl-4-hepten-3-one obtained by condensation of 2-butanone with isobutyaldehyde. The enamine and 6-methyl-4-hepten-3-one were heated under reflux in toluene for 4 days. Preliminary analysis of the complex crude reaction product indicated some delta-cadinenone was formed. Following isolation, this will be reduced in two steps to delta-cadinene.

The total synthesis of delta-cadinene is reported to give an isolated yield of less than 1%. This is less than the amount of delta-cadinene in Cade Oil. Accordingly, isolation by preparative gas chromatography is being carried out.

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